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TECHNICAL REPORT NO. 3

Cathodic Electrochromism of Lutetium Diphthalocyanine Films

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Prenared for Publication

in the

Journal of the Electrochemical Society



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Autonetics Strategic Systems Division Rockwell International Anaheim, California

March 1981

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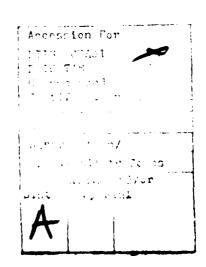
### CATHODIC ELECTROCHROMISM OF LUTETIUM DIPHTHALOCYANINE FILMS

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### **ABSTRACT**

Cathodic electrochromism of lutetium diphthalocyanine films on insulating substrates was investigated by a moving-boundary technique. Reduction occurred by injection of electrons from a gold contact and cations from a liquid electrolyte. Light blue products formed with aqueous or organic solutions of alkali metal salts, while a dark violet product formed with aqueous hydrochloric acid. The dark material had a charge-carrier mobility of  $8 \times 10^{-7} \, \text{cm}^2/\text{V-sec}$  and an estimated bulk resistivity of 1,800 ohm-cm. Reduced forms of the dye appear to be solid cation conductors.



\*Flectrochemical Society Active Member.

Key words: diphthalocyanines, electrochromism, cathodic reduction, cations.

Previous papers from this laboratory have reported investigations of anodic processes in lutetium diphthalocyanine films (1-4). Generally, these are 2-electron oxidations in which the color of the dye changes from green to red. The electrochromic reaction was shown to occur with migration of anions from the aqueous electrolyte into the organic solid. With a chloride solution, it is represented by

$$LuH(Pc)_{2} + 2C1^{-} \rightarrow LuH(Pc)_{2}^{++} \cdot 2C1^{-} + 2e$$
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A solid-state moving-boundary technique was used to determine the faradaic n values, ionic mobilities, and approximate bulk resistivities in the red phases formed with chloride and sulfate ions (1). The anion mechanism was confirmed in a subsequent tracer study (2), and its dependences on ambient water vapor and oxygen were reported recently (4).

Lutetium diphthalocyanine also undergoes cathodic reactions. As a result, it is a multicolor electrochromic material of considerable interest for display applications (5-7). Colors of the reduced films can range from blue-green through blue, dark blue, and violet. Reference 5 gives absorption spectra recorded at 0.1-V intervals on a tin-oxide supported film under essentially open-circuit conditions in both anodic and cathodic ranges. Spectral characteristics as well as Munsell and CIE color coordinates are summarized in References 5 and 6.

 $<sup>^*</sup>$ H<sub>2</sub>Pc is the usual abbreviation for metal-free phthalocyanine; Pc $^{\pm}$  is the anion  $C_{32}H_{16}N_8^{\pm}$ , and LuH(Pc)<sub>2</sub> is commonly called lutetium diphthalocyanine.

This paper describes the results of cathodic moving-boundary experiments on lutetium diphthalocyanine films supported by insulating substrates. The electrolytes were aqueous HCl, aqueous KCl, KI in acetonitrile (AN), and LiCl in dimethyl sulfoxide (DMSO). Again, the solid reaction products appeared to be ionic conductors. The propagated color was light blue with the metal salt solutions and violet with HCl.

### Experimental

Lutetium diphthalocyanine films were prepared by vacuum sublimation of the dye onto sapphire or Mylar strips 1.25 cm wide and approximately 5 cm long. The weight of dye per cm $^2$  was determined from the optical density (OD) of the green film at the 670-nm absorption maximum. The geometric film thickness was estimated from this weight and the approximate crystal density. It ranged from 530 to 1,060 Å. Details of the dye synthesis and specimen preparation were given previously (1). The other materials were reagent-grade chemicals. The organic solvents were dried by allowing them to stand over a molecular sieve.

A sputtered gold contact was applied at the upper end of the dye film, and the lower end was immersed in the liquid electrolyte. The counter electrode was silver foil. A drawing of the moving-boundary cell is given in Reference 1. The cell was assembled in a helium-atmosphere glove box, where the oxygen level did not exceed a few ppm. Constant cathodic currents of 0.2 to 2 µA were applied with a PAR 173 galvanostat, and the total applied voltage was monitored with a Keithley 61°C electrometer and a strip-chart recorder. The boundary-propagation distance was measured by viewing the film against a back-lighted screen of millimeter graph paper under ~2X magnification. The

electric field in the color-converted film could be determined by raising the electrolyte level in successive increments and observing the corresponding voltage decreases. In several cases, the specimens were examined after propagation by energy-dispersive x-ray spectroscopy (EDS), using an ETEC Autoscan scanning electron microscope. Comparisons of lutetium, potassium, and chlorine levels in different regions of the films were made in this way.

## Results and Discussion

<u>Visual Observations.</u>— The color chance always began at the dye/electrolyte interface and travelled upward toward the electronic contact. This behavior is consistent with an ion-injection mechanism. It differs from that of Yamana's qualitative experiments, wherein the blue coloration of erbium diphthalocyaniae films on glass began at the metal contact (8). In our experience, color-converted diphthalocyanine films are prone to crack and peel away from glass. If this occurred in Yamana's cell, the dye could have become wet with electrolyte by capillary action. Cathodic reaction then would have occurred in the upper part of the film.

The adhesion problem was mitigated, though not fully avoided, in the present work by using a sapphire or Mylar substrate.\* With aqueous electrolytes, the cathodic boundary propagation tended not to proceed as uniformly as it did for the anodic green-to-red process (1), but the boundary velocity and field were measurable in a number of experiments, as shown by Fig. 1-3. Occasionally, the boundary was too irregular for quantitative use.

The dye film adheres well to tin oxide, but that substrate cannot be used in moving-boundary studies because it is electronically conductive.

An additional difficulty arose with the organic electrolytes. Although the initial green form of the dye was insoluble in several organic liquids, including AN and DMSO, the blue reduced form was soluble. This led to poor electrical contact during the boundary propagation and prevented determination of the field by changing the electrolyte level.

The propagated color depended on the electrolyte cation. With HCl, the product was dark violet. With KCl, KI, and LiCl, only a light blue appeared. In contrast, we have observed that anodic boundary propagation with different anions yields a single red color (4).

<u>Electrical Parameters.</u>—The results for aqueous and nonaqueous solutions are summarized in Table I. Although the total applied currents were in the microampere range, the estimated current densities through the cross sections of the films were 15 to 200 mA/cm<sup>2</sup>. This range includes current densities that would be used in potentiostatic switching of a diphthalocyanine electrochromic display (7).

The apparent number n of electrons transferred per molecule of dve was calculated from the current, the area converted per unit time, and the optical density of the green film. Plots for evaluating the boundary velocity are shown in Fig. 1 and 2. With aqueous KCl, n was 1.9 · 0.2. With HCl, the apparent n was considerably larger, and on Specimen 1, it was shown to increase systematically with the applied current. The large apparent n's in the acid solution probably were caused by a side reaction forming elemental hydrogen. The apparent n at 0.4 mA was 5.2 for Specimen 1 and 12 for Specimen 2. One could surmise from this comparison that the sapphire substrate was more conducive than Mylar to proton transport beneath the dye film, but further investigation would be needed to verify such a mechanism. Extrapolation of the apparent n to zero current yielded a value of 3.5 for

the Mylar-supported film contacted by 1.2 M HCl. Fewer data were recorded for nonaqueous electrolytes because of the blue-solubility problem, but an n value of 1.1 was obtained with both Mylar- and sapphire-supported films contacted by LiCl-DMSO.

A carrier mobility  $\boldsymbol{\mu}$  in the converted film was found from the relationship

$$\mu = v/\epsilon \tag{1}$$

where v is the boundary velocity and & is the electric field determined by raising the electrolyte level (1). Figure 3 shows plots for the evaluation of & in the Mylar-supported film propagated from aqueous HC1. The average mobility was  $8 \times 10^{-7} \text{ cm}^2/\text{V-sec}$ . Since the current and the approximate film thickness were known, the bulk resistivity  $\rho_b$  of the blue phase could be estimated from Eq. [2], where I is the current density.

$$p_b = 8/1 \tag{2}$$

The average resistivity for Specimen 1 was 1,800 ohm-cm. This is similar to the resistivities of 1,300 and 2,000 ohm-cm for the red oxidation products containing chloride and sulfate ions (1).

<u>Interpretations</u>.-- The violet phase propagated from an aqueous HCl interface appears to be an ionic conductor. This is suggested by the magnitude of the carrier mobility, which is compared in Table II with those of related solid

electrolytes. Dependence of the reduction-product color on the electrolyte cation also is consistent with a cation-injection mechanism. Moreover, EDS analysis confirmed the presence of potassium, and the absence of chlorine, in blue areas propagated from KCl, while neither of these elements was found in green areas of the same plates.

The reaction for aqueous KCl may be written

$$LuH(Pc)_{2} + 2K^{+} + 2e \cdot LuH(Pc)_{2}^{-} \cdot 2K^{+}$$
Green
Light Blue

This process is analogous to the anodic oxidation utilizing two chloride ions and forming the divalent organic cation.

The measurements with LiCl in DMSO indicated a 1-electron reduction, which might be represented by

$$LuH(Pc)_{2} + Li^{+} + e + LuH(Pc)_{2}^{-} \cdot Li^{+}$$
Green
Light Blue

With dissolved lutetium diphthalocyanine, Corker, Grant, and Clecak also reported cathodic formation of a radical anion (11). The electrolyte was tetrabutylammonium fluoborate in dimethylformamide, and the working electrode was platinum. Reduction beyond the 1-electron stage with LiCl in the moving-boundary cell might have been prevented by separation of the two phthalocyanine rings to form LiHPc and LuPc. Such a process could account for the higher solubility in organic solvents.

Further work is needed to characterize the reduction products. It is significant, however, that dark blue or violet colors never were obtained with metal cations in the moving-boundary experiments, even at a current density as high as 200 mA/cm<sup>2</sup>. An acidic electrolyte is required to form the dark product in insulator-supported films. Yet dark blue and violet are readily obtained

with aqueous KCl when the dye is on semiconductive tin oxide (5,6). We propose the following mechanisms to account for this difference in the electrochromic behavior.

With an adequate supply of protons, reduction of the dye can proceed by a double-injection mechanism similar to that of Reaction II:

All or part of the n+1 hydrogen atoms may be ionized in the solid state. When the supply of protons is insufficient, the dye may be converted at a tin oxide electrode by a coupled chemical reaction rather than direct electron transfer:

$$H_2O + e \rightarrow H \cdot (Ads) + OH^-$$

The reactive intermediate is represented as an adsorbed hydrogen atom because a species with such characteristics is known to form on tin oxide at cathodic potentials (12,13). Furthermore, a pH increase accompanies cathodic formation of a blue product on tin oxide in aqueous KCl, and hydrogen ions are released to the solution on its anodic oxidation (14).

The dark blue and violet forms cannot yet be distinguished on the basis of the n values. One of our previous investigations provides evidence, from correlation of absorption spectra with approximate open-circuit potentials of the reduced films, that the dark blue and violet colors may involve two discrete oxidation states of the dye (5). We now estimate that n is 2 or 3 for the dark blue state, and 3 or 4 for the violet.

Moskalev and Kirin originally proposed field-induced ionization to  $\operatorname{H}^{\dagger}$  and  $\operatorname{Lu}(\operatorname{Pc})_2^-$  to account for the cathodic electrochromism of lutetium diphthalocyanine

films (15). The present study has shown that these color changes are due, instead, to faradaic processes. Although the acidic hydrogen may not be firmly bound to the organic structure in rare-earth diphthalocyanines (16), it is the electrolyte cation that plays a vital role in the electrochromism at cathodic potentials.

# Acknowledgment

This work was supported in part by the Office of Naval Research.

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Table I. Results of Cathodic Boundary Propagation in Lutetium Diphthalocyanine Films

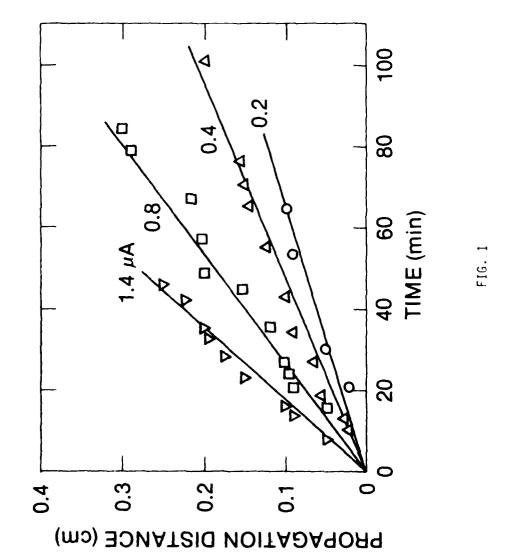
(cm <sup>2</sup> /V-sec) × 10 <sup>7</sup>		;	!	;	1	;	;	1
Apparent n	8.7.7.0 9.00 9.00	12	1.7	2.1	1.s	(r)	7.	
Color Formed	Violet	Violet	Light blue					
Current (:.A)	0 0.2 0.4 0.3	0.4	0.5		2	0.5	0.5	0.5
Electrolyte	1.2 M HCl	1 M HC1	1 M KC1	1 M KC1	1 M KC1	0.1 M KI	~2M LiC1	~2M Lici
Solvent	Water	Water	Water	Water	Water	AN	DWSO	DMSO
Substrate	Mylar	Sapphire	Sapphire	Sapphire	Sapphire	Sapphire	Mylar	Sapphire
Initial OD at 670 nm	1.15	1.02	0.82	0.85	0.85	69.0	0.58	1.02
Specimen	_	2	m	4	5	9	7	8

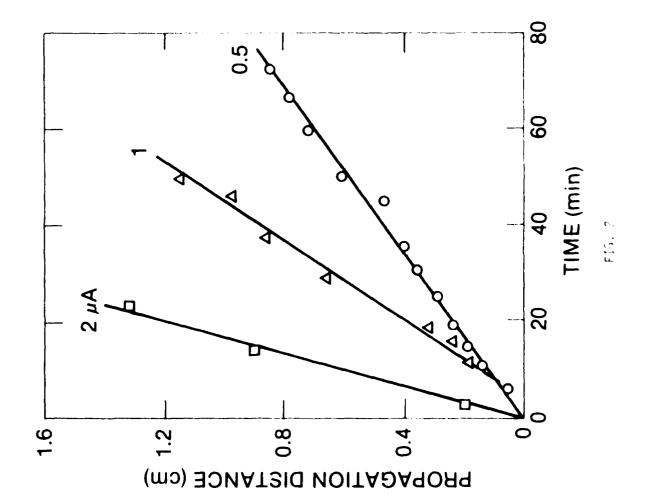
\*By extrapolation of n vs i to i = 0.

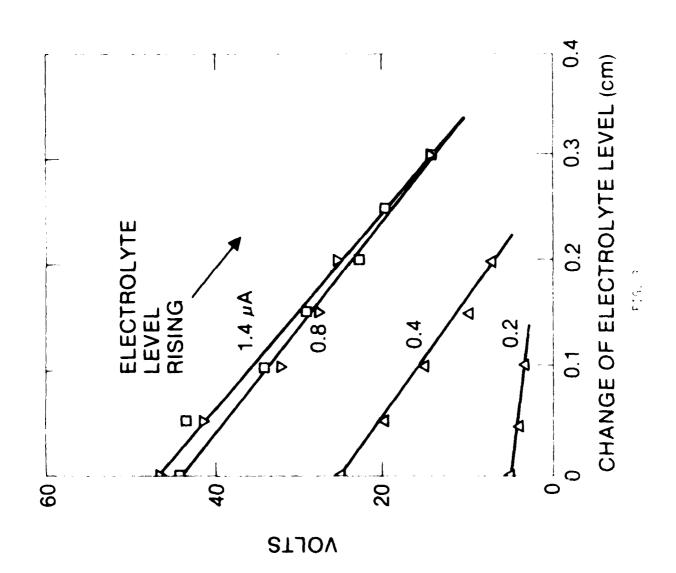
This investigation Reference 10  $\boldsymbol{\varphi}$ Mobility (cm2/V-sec) x 106 Table II. Solid-State Ionic Mobilities Near 25 0.8 0.8  $\infty$ Mobile Ion c1 = 504 Polycrystalline Form Thin film Thin film Thin film Anodic oxidation product of LuH(Pc)<sub>2</sub> Cathodic reduction product of LuH(Pc)<sub>2</sub> Material  $\mu 100_2 \rho 0_4.4 \mu_2 0$  $H_{x}W0_{3}$ 

### Illustrations

- 1. Dependence of boundary propagation distance on time with aqueous 1.2  $^{\rm M}$  HCl electrolyte. Specimen 1.
- 2. Dependence of boundary propagation distance on time with aqueous 1  $^{\rm M}$  KCl electrolyte. Specimens 3, 4, and 5.
- Plots for determination of electric field in the blue film with aqueous 1.2 M HCl electrolyte. Specimen 1.







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